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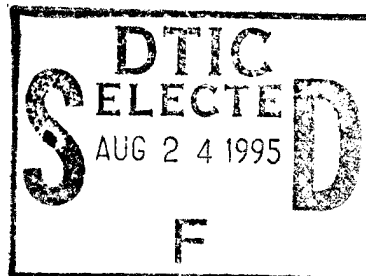


Solid State Lithium/Polymer Battery

Charles W. Walker, Jr.

ARL-TR-429

June 1995



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REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188	
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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE June 1995	3. REPORT TYPE AND DATES COVERED Technical Report: Jul to Dec 93		
4. TITLE AND SUBTITLE Solid State Lithium/Polymer Battery		5. FUNDING NUMBERS		
6. AUTHOR(S) Charles W. Walker, Jr.				
7. PERFORMING ORGANIZATION NAMES(S) AND ADDRESS(ES) US Army Research Laboratory (ARL) Physical Sciences Directorate (PSD) ATTN: AMSRL-PS-CC Fort Monmouth, NJ 07703-5601		8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR-429		
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSORING / MONITORING AGENCY REPORT NUMBER		
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.		12 b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) An all-solid-state lithium rechargeable cell has been fabricated with an ionically conductive solid polymer electrolyte (SPE) and an electronically conductive polymer cathode. The SPE was comprised of lithium salts (LiCF ₃ SO ₃ , LiN(CF ₃ SO ₂) ₂) dissolved in poly(ethylene oxide) (PEO). The polymer cathode, poly(3-methylthiophene) (PMT), was formed by electropolymerization. Cell discharge/charge reactions involve lithium stripping/plating at the anode and anion undoping/doping at the cathode. Lab cells cycled for up to 170 cycles at temperatures between 19°C and 22°C exhibited good capacity retention.				
14. SUBJECT TERMS Batteries; lithium; nonaqueous electrolytes; cathodes; polymers; thiophenes; rechargeable		15. NUMBER OF PAGES 20		16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

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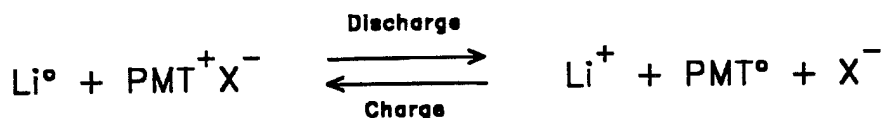
INTRODUCTION

Lithium primary batteries are used extensively in military applications because they can provide high power and high energy density over a wide range of temperatures. Lithium batteries are slowly gaining a place in commercial markets as well, primarily for low current applications (to ensure maximum safety). Because the lithium-based systems are expensive, a rechargeable lithium chemistry is needed in order to be cost-competitive with other battery chemistries. The approach described here involves the use of an electronically conductive polymer which functions as the cathode, and an ionically conductive solid polymer electrolyte. This system is rechargeable and does not contain a liquid electrolyte, eliminating problems associated with electrolyte leakage and corrosion of metallic lithium.

As an electrolyte, there are many advantages associated with the use of freestanding, ionically conducting polymers (e.g., PEO) including the use of high speed film processing technology to construct thin, lightweight batteries of any desired size and geometry. A solid electrolyte is comprised of a polymer host containing dissolved lithium salt(s), allowing the movement of Li^+ and X^- ions between the electrodes. Batteries can be sealed in metallized plastic film pouches, eliminating the weight and volume associated with metal cans that are required when using liquid electrolytes. There is also a safety advantage in that there is no liquid electrolyte to leak from cells. In addition to its role as an electrolyte, an SPE film can also act both as a mechanical separator between the anode and cathode, and as a binder/adhesive to insure contact between electrodes. Elasticity allows the SPE to conform to electrode volume changes during cycling.

Polymer cathodes composed of thin electropolymerized films can be prepared which, when doped with anions, are electronically conductive. Upon discharge, anions return to the electrolyte and the film becomes nonconductive. Micron-thick films have been used successfully as cathodes in lithium cells containing organic and inorganic electrolytes [1-4].

Thin, all-solid-state Li/SPE/PMT lab cells were constructed and cycled. The PMT electrode accepts anions common to the SPE on charge, and releases anions into the SPE upon discharge. At the lithium electrode, lithium ions from the SPE are plated as metallic lithium during charge, and released to the SPE on discharge (see below). Since anions and cations are simply being shuttled between



electrodes, and no new products are formed during charge or discharge, no structural damage or chemical reactions detrimental to cycle life should occur. For these reasons, exceptional cycle life and capacity retention are anticipated.

EXPERIMENTAL

Polymer electrolyte was prepared by dissolving PEO (Aldrich, MW = 4×10^6 , dried at 50°C under vacuum overnight), LiCF_3SO_3 (Aldrich, 97%, dried 110°C under vacuum), and $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ (3M, 99.5%, dried 110°C under vacuum) in a molar ratio of 20:1:1 in acetonitrile (Fluka, 99.5%, distilled under a stream of dry argon), to form a viscous solution. A binary salt system was chosen because PEO films containing either salt alone are unsuitable. Although freestanding films containing only LiCF_3SO_3 are mechanically strong, ionic conductivity is low ($\sigma \approx 5 \times 10^{-7} \text{ S cm}^{-1}$). PEO containing only $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ shows much higher conductivity ($\sigma \approx 5 \times 10^{-5} \text{ S cm}^{-1}$) due to the ability of this salt to plasticize PEO, however, this film composition is soft and very tacky. It will readily stick to itself, unable to be pulled apart. By combining both salts in a 50:50 ratio, we found the binary salt formulation produced a mechanically rugged, easily handled freestanding film with an ionic conductivity only slightly lower than that of the film containing $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ salt alone [5].

PMT films were electrochemically polymerized on a platinum rod substrate (0.071 cm^2) from solutions containing 0.1 M 3-methylthiophene monomer (Sigma Chemical, 99+%) dissolved in CH_3CN which also contained a 0.1 M concentration of either tetrabutylammonium tetrafluoroborate (Alpha, recrystallized with acetone and ether, dried at 105°C under vacuum), LiCF_3SO_3 or $\text{LiN}(\text{CF}_3\text{SO}_2)_2$. The exact procedure for the polymerization of 1.4 μm thick cathodes has been described in detail previously [1-4]. The resulting films contained dopant anions (BF_4^- , CF_3SO_3^- or $\text{N}(\text{CF}_3\text{SO}_2)_2^-$) corresponding to the salt used. Based on film thickness and cross-sectional area, the volume of the PMT was $9.95 \times 10^{-6} \text{ cm}^3$. Assuming the ideal case (100% efficiency) during electropolymerization, a maximum of $4.52 \times 10^{-5} \text{ g}$ of PMT was deposited. Because the true mass was certainly less, the Ah g^{-1} calculated for actual cell discharges is low.

PMT-covered platinum electrodes were fitted with a small lithium metal reference electrode (Figure 1), then dipped into the polymer solution four or five times. Between each dip, films were allowed to stand to permit the CH_3CN to evaporate, leaving solid polymer electrolyte covering the PMT and reference electrodes. Finished electrodes were dried overnight under active vacuum at 50°C. Laboratory cells were constructed by pressing electrodes against metallic lithium (anodes) and maintaining slight pressure to ensure mechanical contact. Cell cycling was performed galvanostatically with an EG&G PAR Model 173 potentiostat/galvanostat controlled by a HP86B computer. All preparations and testing

were performed in a glove box containing a dry (<2 ppm H₂O) argon atmosphere.

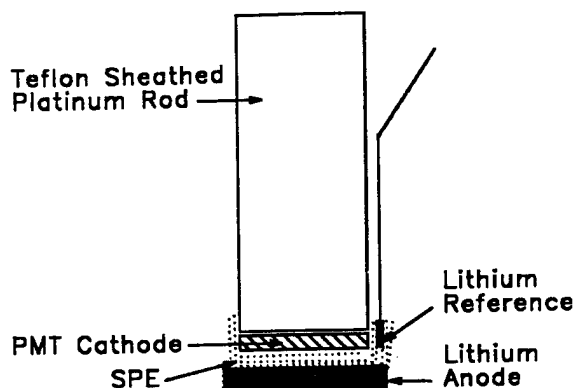


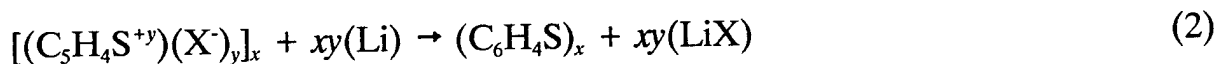
Figure 1. Schematic of laboratory cell.

RESULTS AND DISCUSSION

During cell discharge PMT is reduced (undoped). Theoretical energy density obtained from undoping depends on the molecular weight of the dopant anion and level of doping, and can be calculated by

$$\frac{Ah}{kg} = \frac{(96480 \text{ C/F})(1000 \text{ g/kg})}{(g/F)(3600 \text{ s/h})} = \frac{26800}{(g/F)} \quad (1)$$

Writing the overall discharge reaction and solving for g/F by inserting the formula weights for the electroactive species we have:



where X⁻ is the anion and y represents the percent doping.

$$g/F = \frac{x(C_5H_4S) + xy(LiX)}{xy} \quad (3)$$

$$g/F = \frac{x(96.15) + xy(\text{f.w. of LiX})}{xy} \quad (4)$$

$$g/F = \frac{96.15}{y} + \text{f.w. of LiX} \quad (5)$$

It is clear that energy density will increase with increasing dopant levels and with decreasing formula weight of the anion. Figure 2 shows theoretical energy density (based on the ability to fully undope) vs dopant level for a series of anions. Undoped PMT is electrically insulating, so the theoretical energy cannot be realized since PMT could never become fully undoped.

Increasing the level of doping does not necessarily increase conductivity. Ofer *et al.* [6] determined that there is a maximum in conductivity beyond which conductivity decreases with further doping. For PMT they found a maximum in conductivity occurred at ≈ 0.3 electrons per repeat unit, and beyond ≈ 0.5 electrons per repeat unit thiophenes tended to decompose. Other evidence arguing for doping levels of about 30% was shown in studies with poly(3-octylthiophene) by Kawai *et al.* [7]. They determined a coulombic efficiency of only 47% at a maximum doping level of 48.6%, whereas at a 33% doping level efficiency was 94%.

Cell Type: $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)_1(\text{LiN}[\text{CF}_3\text{SO}_2]_2)_1/\text{PMT-BF}_4^-$.

In this example, PMT was polymerized with BF_4^- as the dopant anion. This cell was discharged at $5 \mu\text{A cm}^{-2}$ to a 2.0 V cutoff, and charged at $2.5 \mu\text{A cm}^{-2}$ to a 3.8 V cutoff for 170 cycles at a temperature between 19°C and 22°C . Initial load potential was above 3.6 V, with voltage gradually decreasing until near the end of discharge when voltage dropped abruptly from about 3.0 V to the 2.0 V cutoff.

Although theoretical energy density of the PMT electrode is highest with this anion (see Figure 2), SPE containing LiBF_4 is a poor ionic conductor. It was supposed that initial capacity would be high, but with cycling it would drop somewhat as PMT would be doped with a mixture of BF_4^- , CF_3SO_3^- , and $\text{N}(\text{CF}_3\text{SO}_2)_2^-$. However, this was not the case. Good retention of capacity was observed (Figure 3), and mean cycling efficiency was 98.4% (Figure 4). The increase in capacity over the first few cycles is explained by an increase in the level of polymer doping as the PMT becomes better able to accommodate anions. As polymerized, PMT films contain less than optimal levels of dopant.

Cell Type: $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)_1(\text{LiN}[\text{CF}_3\text{SO}_2]_2)_1/\text{PMT-CF}_3\text{SO}_3^-$.

The CF_3SO_3^- dopant anion introduced during PMT electropolymerization created a cell in which the dopant was common to anions present in the electrolyte. With cycling, a mixture of CF_3SO_3^- and $\text{N}(\text{CF}_3\text{SO}_2)_2^-$ anions will dope the PMT. Discharge ($5 \mu\text{A cm}^{-2}$ to 2.0 V) and charge ($3.5 \mu\text{A cm}^{-2}$ to 3.8 V) capacity increased over the first few cycles from 2 mAh g^{-1} to 3.5 mAh g^{-1} until cycle 19, where capacity suddenly dropped to 1.2 mAh g^{-1} (Figure 5). Thereafter, capacity was consistent, fading to slightly above 1.0 mAh g^{-1} by cycle 87.

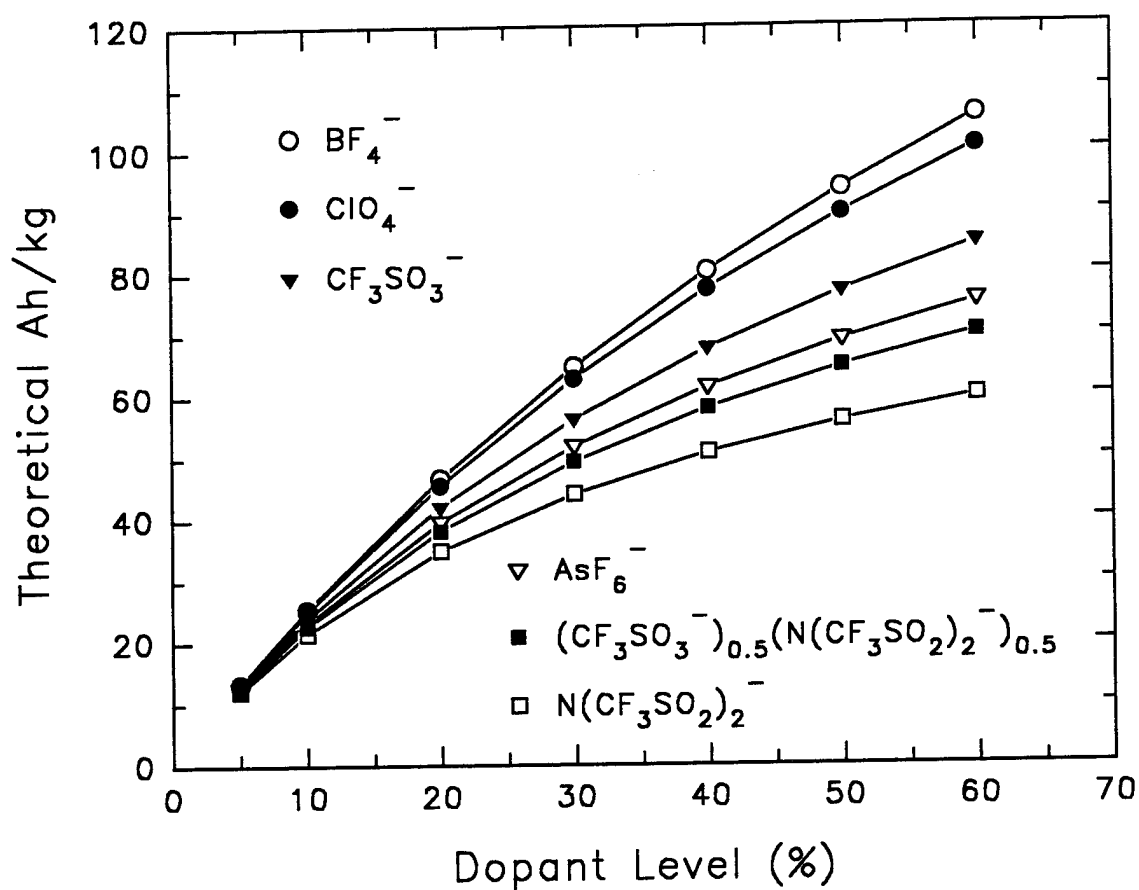


Figure 2. Theoretical energy density as a function of level of doping for PMT doped with various anions.

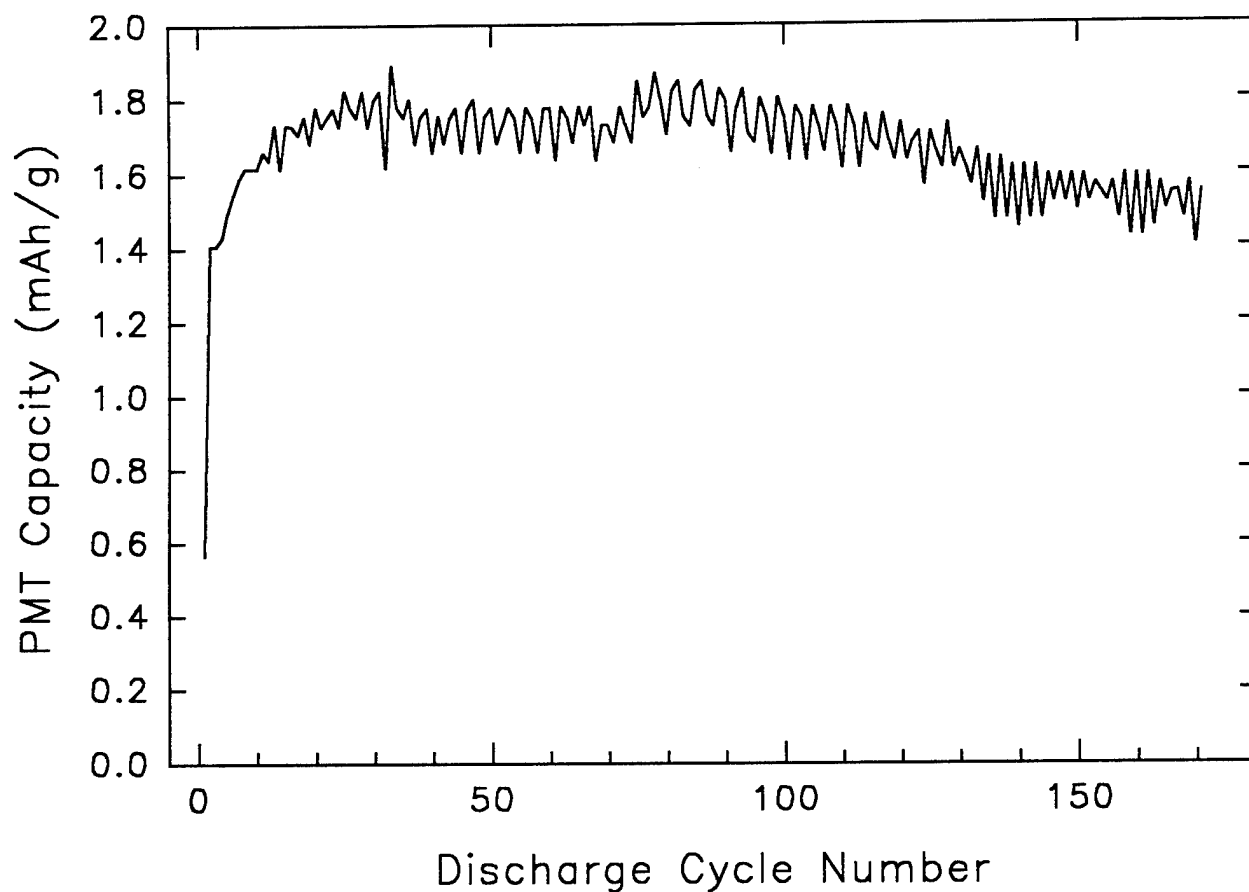


Figure 3. Capacity of $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)(\text{LiN}[\text{CF}_3\text{SO}_2]_2)/\text{PMT}^+\text{BF}_4^-$ cell at 20°C . Discharge rate of $5 \mu\text{A cm}^{-2}$ to 2.0 V, charge rate of $2.5 \mu\text{A cm}^{-2}$ to 3.8 V.

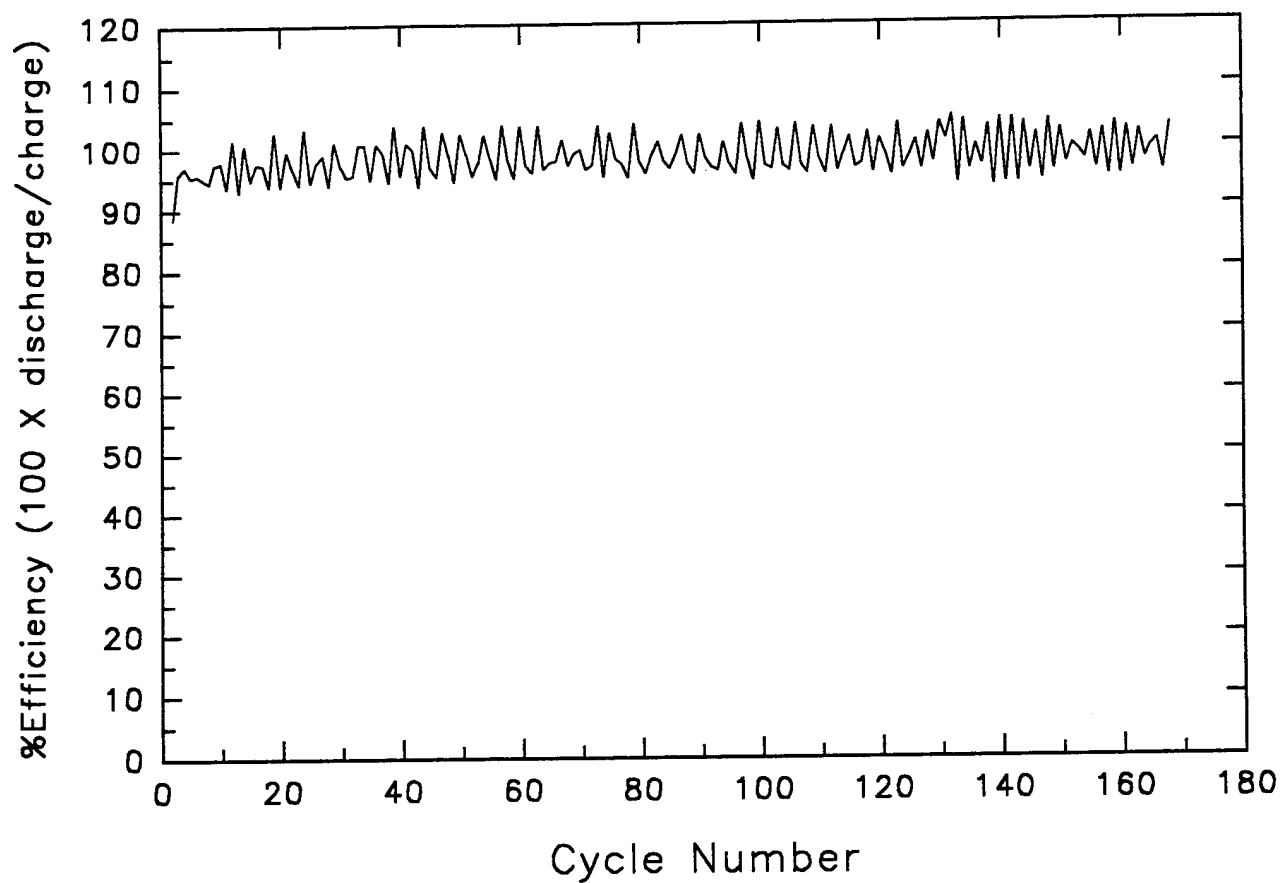


Figure 4. $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)(\text{LiN}[\text{CF}_3\text{SO}_2]_2)/\text{PMT}^+\text{BF}_4^-$ cell cycling efficiency at 20°C .

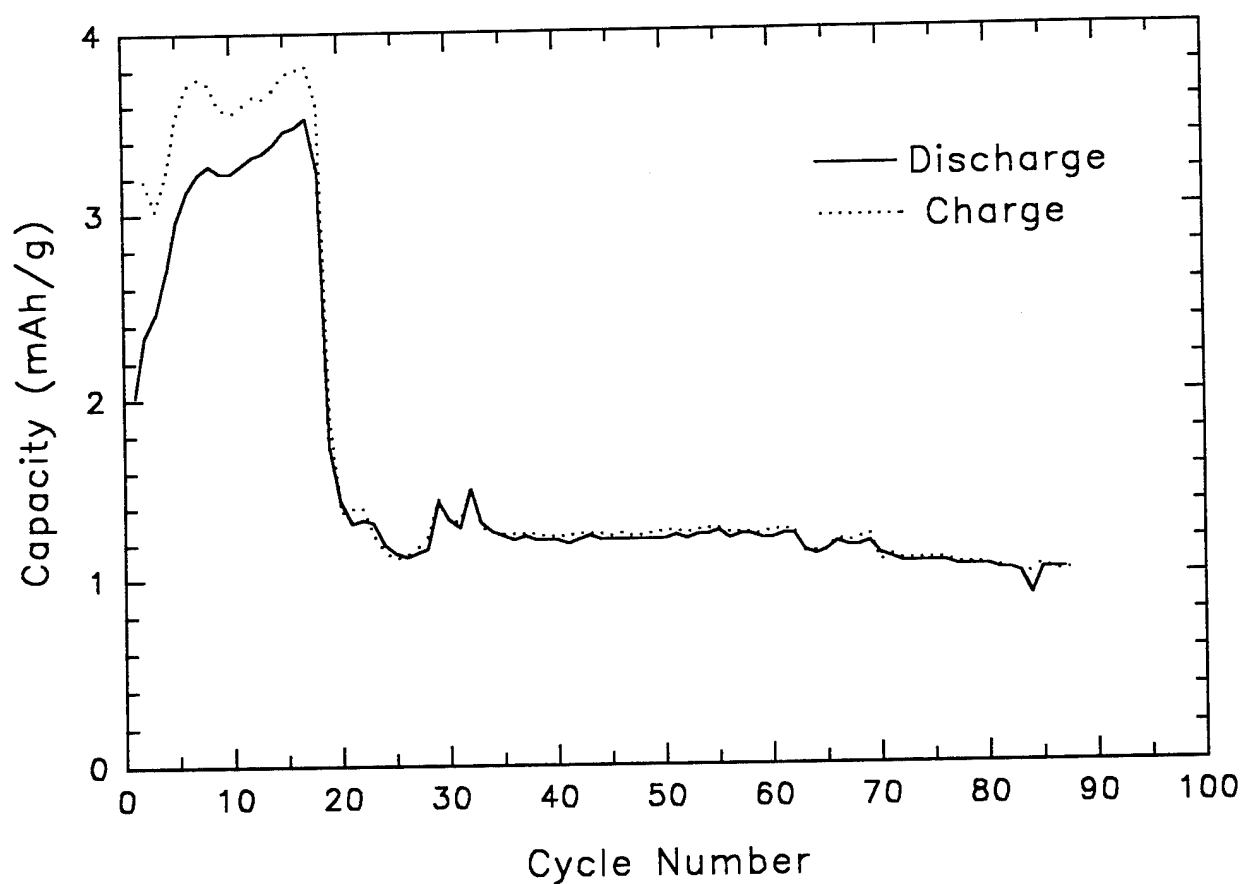


Figure 5. $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)(\text{LiN}[\text{CF}_3\text{SO}_2]_2)/\text{PMT}^+\text{CF}_3\text{SO}_3^-$ cell capacity at 20°C. Discharge (solid line) at $5 \mu\text{A cm}^{-2}$ to 2.0 V, charge (dotted line) at $2.5 \mu\text{A cm}^{-2}$ to 3.8 V.

A second cell was cycled at $5 \mu\text{A cm}^{-2}$ rate for both discharge and charge (Figure 6). Initial capacity was much greater than the first cell, indicating significant cell to cell variation which can be attributed to an inconsistent cell assembly technique. Capacity of this second cell was initially above 9 mAh g^{-1} , abruptly dropping to 6 mAh g^{-1} at cycle 18. The reason for this sharp drop in capacity is unknown, but was common to both cells after about the same number of cycles. A similar event also occurred in a cell (described below) containing PMT doped with the $\text{N}(\text{CF}_3\text{SO}_2)_2^-$ anion.

Cell Type: $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)_1(\text{LiN}[\text{CF}_3\text{SO}_2]_2)_1/\text{PMT-N}(\text{CF}_3\text{SO}_2)_2^-$.

In this example, the imide anion $(\text{N}(\text{CF}_3\text{SO}_2)_2^-)$ initially doped into the PMT was also common to the electrolyte. Discharge and charge were both at a rate of $5 \mu\text{A cm}^{-2}$ between voltage cutoffs of 2.0 V and 3.8 V, respectively. Capacity increased over the first few cycles (Figure 7) to 12.9 mAh g^{-1} . At cycle 25, capacity fell to 6.6 mAh g^{-1} then recovered slightly to remain between 7.5 to 8.5 mAh g^{-1} through cycle 72.

At this point acetonitrile vapors were present within the glove box and were probably absorbed into the SPE as evidenced by the sharp rise in capacity ($\sim 23 \text{ mAh g}^{-1}$). This could be explained as increasing solvation of the salt, permitting a better ionic transport within both the SPE and PMT. This result indicates that introducing a small amount of plasticizing solvent such as acetonitrile into the SPE produces a great increase in capacity (which would be retained in a sealed cell). This is reasonable, based on previous work [2] where PMT was used with an organic electrolyte, averaging about 54 mAh g^{-1} . Here, capacity was extremely high by comparison, due to two factors: higher electrolyte conductivity and greater ionic mobility.

After charging, the cell sat 4.5 days at open circuit before cycling was continued. During this period, the acetonitrile was presumably adsorbed by fresh molecular sieves introduced into the box to collect the vapors. Initially, capacity declined to 6 mAh g^{-1} but then returned to nearly 8 mAh g^{-1} . Apparently, charge retention on open circuit is not very good, but the capacity losses can be recovered with subsequent cycling.

CONCLUSIONS

Based on these preliminary studies, it is clear that energy densities of 12 mAh g^{-1} are possible in a solid state lithium rechargeable cell containing an ionically conductive solid polymer electrolyte and electronically conductive polymer cathode. Because the cell assembly technique was not optimized in this study, it can not be determined which, if any, anion is the preferred dopant for PMT. If a small amount of plasticizing solvent is added to the SPE, much higher capacities could be expected since ion mobility would increase.

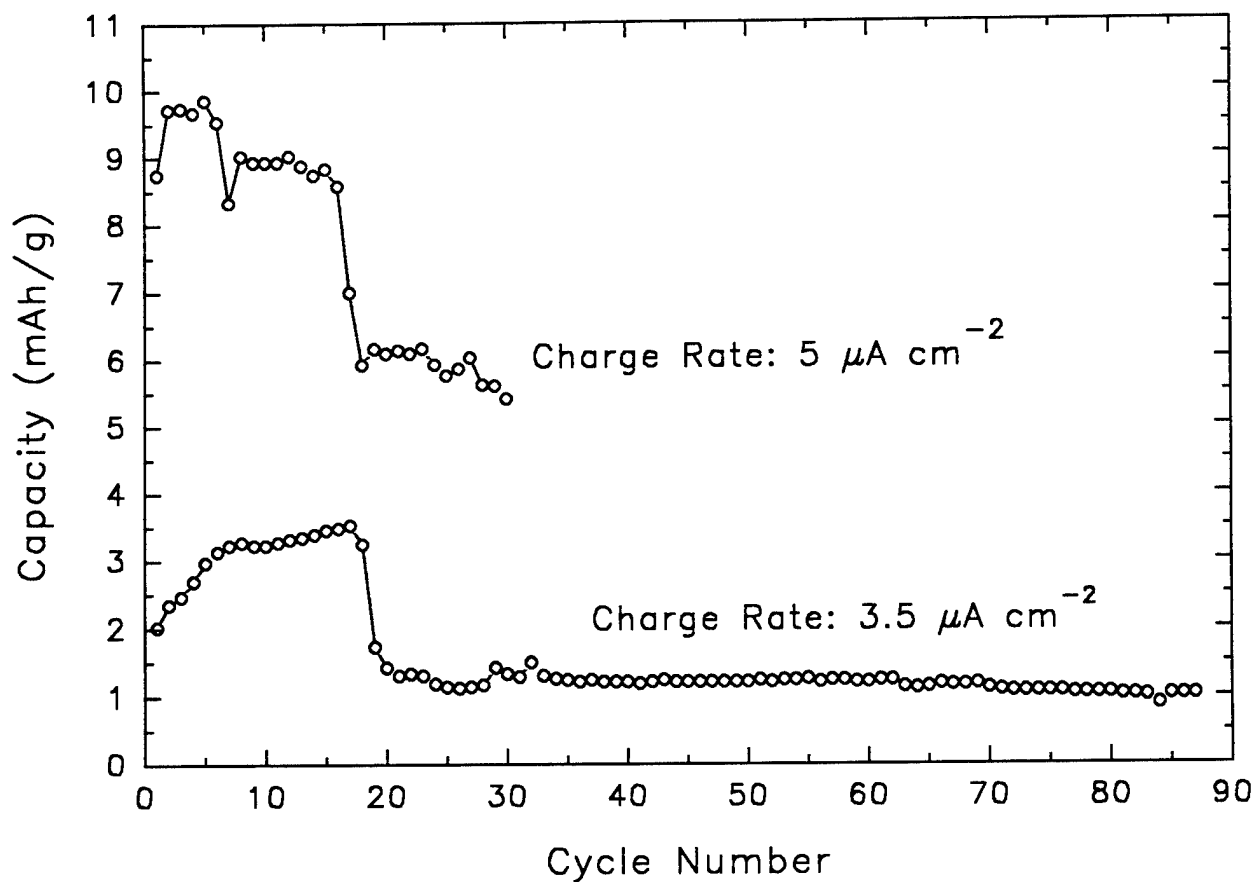


Figure 6. $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)(\text{LiN}[\text{CF}_3\text{SO}_2]_2)/\text{PMT}^+\text{CF}_3\text{SO}_3^-$ cell capacity at 20°C , comparing two cells discharged at $5 \mu\text{A cm}^{-2}$ to 2.0 V, and charged at either $3.5 \mu\text{A cm}^{-2}$ or $5 \mu\text{A cm}^{-2}$ to 3.8 V.

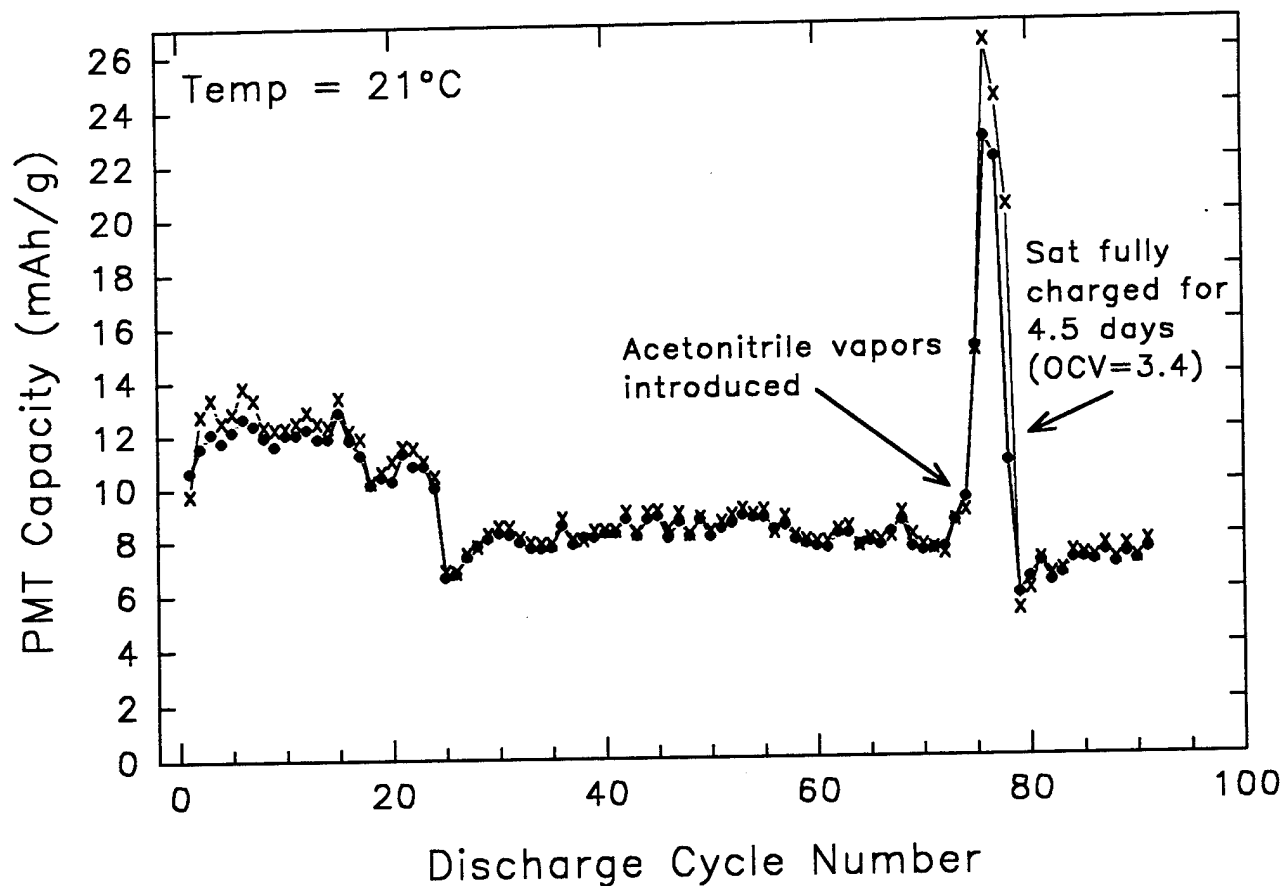


Figure 7. $\text{Li}/(\text{PEO})_{20}(\text{LiCF}_3\text{SO}_3)(\text{LiN}[\text{CF}_3\text{SO}_2]_2)/\text{PMT}^+\text{N}(\text{CF}_3\text{SO}_2)_2^-$ cell capacity at 20°C. Discharge (●) at $5 \mu\text{A cm}^{-2}$ to 2.0 V, charge (x) at $5 \mu\text{A cm}^{-2}$ to 3.8 V.

In both cells containing PMT doped with an anion common to SPE, there was a sharp drop in capacity after 18 to 25 cycles whereafter capacity stabilized. It is not clear what event could cause an abrupt capacity loss as opposed to a gradual decay in performance as is more common.

One concern when using metallic lithium is the formation of dendrites with repeated cycling that could lead to internal shorting of the cell. An intercalating anode (e.g., Li_xC_6) could provide a greater margin of safety since Li^+ ions are shuttled in and out of the anode, eliminating metallic lithium and the possibility of dendrites. This "lithium-ion" technology would provide the ultimate in safety, and would be amenable to the consumer market.

The system described here could be used to inexpensively construct thin, lightweight, high voltage batteries that are able to be configured in any desired geometric shape. High speed equipment for producing films exists, so it is merely a matter of identifying a suitable chemistry.

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